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(पहला पुनरीक्षण)

Indian Standard

SULPHATED OIL FOR LEATHER FATLIQUORING — SPECIFICATION

(First Revision)

ICS 59.140.10

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

September 2013 **Price Group 4**

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Sulphated oils are used in fatliquoring operation during tanning to provide flexibility to the leather. Fatliquors are generally prepared with sulphated oils, raw oils and other additives.

This standard was originally published in 1971. In this revision, the requirement on pentachlorophenol (PCP) has been introduced keeping in view of the demand for eco-friendly inputs from the leather industry.

The composition of the Committee responsible for formulation of this standard is given in Annex B.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SULPHATED OIL FOR LEATHER FATLIQUORING — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for sulphated oils used in the leather industry for fatliquoring.

1.1.1 This standard covers only sulphated oils of marine and vegetable origin exclusive of any further additives or blends of fatliquors made thereof.

2 REFERENCES

The standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
548 (Part 1) : 1964	Methods of sampling and test for oils and fats: Part 1 Methods of sampling physical and chemical test
1070 : 1992	Reagent grade water (*third revision*)
1640 : 2007	Glossary of terms relating to hides, skins and leather (*first revision*)
4221 : 1967	Quality tolerances for water for tanning industry
14575 : 1999	Determination of pentacholorophenol (PCP) in leather — Method of test

3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 548 (Part 1) and IS 1640 shall apply.

4 REQUIREMENTS

4.1 Physical Condition

The material shall be a free-flowing, clear, oily liquid, appearance of any graininess or a slight physical separation like a liquid and semi-solid portion appearing especially in winter season or long storage shall not be considered as defect so long as the two portions individually or together form ready emulsions in water.

4.2 Emulsion Characteristics

The emulsion shall satisfy the following requirements:

a) The product shall form a ready emulsion in hot or cold water in any dilution.
b) An emulsion containing 3 percent total fatty material shall remain stable for at least 30 min at 27 ± 2°C without creaming or separation.
c) The sulphated oils shall be capable of being readily washed out with water without leaving any oily feeling to hand.
d) The sulphated product when mixed with 25 percent raw oil(s) having an acid value not above 10, or other mineral oil shall form a ready emulsion when diluted with hot/cold water in all dilutions.

4.3 Odour

The product shall be free from rancid or putrefactive odour of the oils.

4.4 Chemical Requirements

The product, besides meeting the other characteristics mentioned above, shall also meet the chemical requirements given in Table 1.

4.5 Keeping Quality

The product shall be stable for at least 6 months when stored in normal packings.

5 PACKING AND MARKING

5.1 Packing

The material after manufacture shall be packed in air-tight containers which shall not develop any rust or deterioration of the product due to the presence of water in the medium or any of the ingredients used in the formulation.

5.2 Marking

The containers shall be marked with the name of the manufacturer, trade-mark, if any; batch number; date of manufacture; and the quantity in litres.

5.2.1 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard mark is governed by the provisions of *Bureau of Indian Standard Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

Table 1 Chemical Requirements

(*Clause* 4.4)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Moisture, percent by mass, *Max*	35	**A-2**
ii)	*p*H of emulsion	6.0 - 8.0	**A-3**
iii)	Total fatty matter, percent by mass, *Min*	60	**A-4**
iv)	Unsaponifiable matter, percent by mass, *Max*	2	**A-5**
v)	Total alkalinity equivalent per 10 g, in mg, *Max*	3	**A-6**
vi)	Organically combined sulphate (SO_3) as sulphuric esters, percent by mass, *Min*	3	**A-7**
vii)	Total organically combined sulphates (SO_3) as sulphuric and sulphonic esters, percent by mass, *Min*	4.5	**A-7** and **A-8**
viii)	Ash, percent by mass, *Max*	3	**A-9**
ix)	PCP content[1)], in mg/kg, *Max*	5	**A-10**

[1)] Calculated on moisture free basis.

ANNEX A

(*Table* 1)

TEST METHODS

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of test or analysis.

A-1.2 Water to be used for tests other than chemical tests (**4.4**, Table 1) shall conform to IS 4221.

A-2 DETERMINATION OF MOISTURE

A-2.1 Outline of the Method

The percentage of water present is determined by distillation with a solvent.

A-2.2 Procedure

Heat 30 g of the oil and 70 ml of a solvent in a 300 ml flask connected, preferably by ground glass connections, to a condenser and a Dean and Stark receiving tube or similar apparatus graduated in divisions of 0.1 ml. Add a few glass beads to the flask to prevent bumping, but conduct the distillation in such a manner that the contents of the flask are in vigorous movement. Continue the distillation until the liquid falling from the condenser is clear, then disconnect the receiving tube, allow to stand and read off the volume of water which has separated from the solvent.

NOTES

1 For sulphated castor oil, toluene or xylene are the preferred solvents. For other oils a light petroleum, fraction boiling between 100 and 140°C is suitable.

2 Thorough cleaning of the apparatus with chromic-sulphuric acid mixture, followed by thorough washing with water prevents the formation of troublesome drops of water difficult to get into the measuring tube.

3 This method is not accurate if the sulphated oil contains mineral acids, free sulphonic acids or free sulphuric esters, alcohol, acetone or other water miscible solvents.

4 If ammonia is present, the specific gravity of the aqueous layer should be determined and taken into account in the calculation.

A-2.3 Calculation

Calculate the percentage of water present in the sample from the reading and the weight of the oil taken for the test.

A-3 DETERMINATION OF *p*H

Determine the *p*H electrometrically, with the help of *p*H meter using glass electrodes.

NOTE — As the *p*H of solution of sulphated oils varies with the temperature, this should be controlled during the tests, and in some cases it may be necessary to make the determination at 50°C.

A-4 DETERMINATION OF TOTAL FATTY MATTER

Follow the procedure given in Method A or Method B.

A-4.1 Procedure

A-4.1.1 *Method A*

If inorganic salts or soaps are present, heat 3-5 g of the sample with 10 ml of 25 percent hydrochloric acid under a reflux condenser until the fat separates out clearly and free from emulsion. Cool and add 50 ml diethyl ether. If any fatty matter is present in the condenser, wash it down into the flask with ether. If after a brief shaking the upper ethereal layer is not clear, repeat the operation with longer heating with hydrochloric acid. Run the solution of fat and the acid wash-water into a separating funnel, run off the acid aqueous layer and when the ether layer is clear, wash with 10 percent sodium chloride (NaCl) until free from acid. If water-soluble fatty acids are present as in oils such as coconut and palm kernel, extract the combined aqueous layers again with ether. If other substances which are split by hydrochloric acid (HCl) only with difficulty are present (alkaline earth soaps, filling materials, etc), repeat the treatment with hydrochloric acid. If emulsions form, which prevent sharp separation, remove the material responsible for the formation of the emulsions by filtration and wash with ether. Dry the combined ether solutions for 30 min over anhydrous sodium sulphate, having first rendered the sodium sulphate fat free by washing with dry ether. Filter the dried ethereal solution of the fat into a weighed flask. Distill off the greater portion of the ether and remove the final traces by blowing over the surface, preferably with a hand blower. The solvent rapidly evaporates and constant weight is rapidly attained by heating at 100°C. The weight may be taken as constant if it falls by not more than 0.1 percent during 15 min heating.

NOTES

1 In the case of fats such as tallow, containing high melting-point fatty acids, it is not convenient to dry off the ether in this manner. The residual water in this case may be removed readily by heating on a water-bath after the addition of 2 to 3 ml acetone to the ethereal solution.

2 If the fat contains volatile fatty acids, for instance, palm kernel, or coconut oil, the-drying is done at 60°C.

3 Easily oxidised fats (drying, semi-drying, fish oils, etc) are dried in a stream of inert gas or in a partial vacuum.

A-4.1.2 *Method B*

Where it is desired to extract the fat without pre-treatment with hydrochloric acid, dissolve 3.5 g of the sample in 10 ml diethyl ether. Dry the ethereal solution over fat-free anhydrous sodium sulphate in a ground glass stoppered flask, to facilitate shaking, for 30 min and filter. If the ether insolubles are required, filter the ethereal solution before drying. Then follow the procedure given in **A-4.1.1.**

NOTE — The ether extract contains the saponifiable fatty matter (neutral fat and free fatty acids) and the other ether soluble materials, such as natural and added unsaponifiable matter, resins, naphthenic acid, and fatty acids combined with bases carried through with the ether in small amounts only.

A-5 DETERMINATION OF UNSAPONIFIABLES

Determine the unsaponifiables following the procedure given in **8** of IS 548 (Part 1).

A-6 DETERMINATION OF TOTAL ALKALINITY

A-6.1 Procedure

Dissolve 10 g of the sample in 100 ml of water in a conical flask, warming if necessary, to obtain solution. After cooling, add 50 g of sodium chloride, 25 ml of ether and 5 drops of 0.1 percent methyl orange indicator and titrate with 0.5 N sulphuric acid until the aqueous layer is orange. Stopper the flask and shake the contents frequently during the titration, and calculate as follows:

$$A = \frac{10\,VN}{W}$$

where

A = total alkalinity equivalent in ml/10 g of oil;

V = volume of sulphuric acid, in ml;

N = normality of sulphuric acid; and

W = weight of the sample, in g.

NOTE — The method is not applicable in the presence of sodium acetate or other compounds which are not accurately titratable to methyl orange in aqueous solution.

A-7 DETERMINATION OF ORGANICALLY COMBINED SO_3 AS NEUTRALIZED SULPHURIC ESTER

A-7.1 Apparatus

A-7.1.1 *Conical Flask, with ground glass neck*, 250 ml capacity.

A-7.1.2 *Splash Head*

A-7.1.3 *Liebig Condenser*

A-7.2 Reagents

A-7.2.1 *Sulphuric Acid,* approximately N, accurately standardized.

A-7.2.2 *Sodium Hydroxide,* approximately N, accurately standardized.

A-7.2.3 *Sodium Chloride*

A-7.2.4 *Diethyl Ether*

A-7.2.5 *Methyl Orange Indicator* — 0.1 percent aqueous solution.

A-7.3 Procedure

Weigh 10 g of the sample into a 250 ml conical flask. Add 30 ml sulphuric acid accurately from a burette, and a few glass beads. Attach splash head and condenser. Boil for 90 min or until both oil and water layers are perfectly clear. Allow to cool. Wash down condenser and splash head with 10 ml water. Disconnect. Add 50 ml water, 30 g sodium chloride (NaCl), 25 ml diethyl ether and 5 drops methyl orange indicator. Titrate with sodium hydroxide with shaking until the aqueous layer is of the same intermediate colour as in the determination of total alkalinity. Run a blank and titrate it also after refluxing.

A-7.4 Calculation

A-7.4.1 Calculate organically combined sulphate (SO_3) existing as neutralized sulphuric esters expressed as miligrams equivalent per 10 g of oil as follows:

Organically combined SO_3 as neutralized sulphuric esters, milligram equivalent per 10 g of oil, $G = A + F$

where

A = total alkalinity in milligrams equivalent per 10 g of oil (*see* **A-6.1**); and

F = increase in acidity on boiling, in milligram equivalent per 10 g of oil and is calculated as follows:

$$F = \frac{(V_1 - V_2)N}{W} \times 100$$

where

V_1 = volume of sodium hydroxide required in the titration of the blank, in ml;

V_2 = volume of sodium hydroxide required in the titration of the sample, in ml;

N = normality of sodium hydroxide solution in N; and

W = weight of the sample taken, in g.

NOTE — If F is negative, retain the sign of the above equation.

A-7.4.2 Calculate organically combined sulphate (SO_3) existing as neutralized sulphuric esters expressed as a percentage on the original sample as follows:

Organically combined sulphate (SO_3) .

as sulphuric esters, percent by mass $= (A + F) \times 0.800\ 66$

$=(A+F) \times 0.800\ 7$

$= G \times 0.800\ 7$

A-8 DETERMINATION OF TOTAL ORGANICALLY COMBINED — SO_3 AS SULPHURIC AND SULPHONIC ESTERS

A-8.1 Determine the organically combined sulphates (SO_3) existing as neutralized sulphuric esters as in **A-7**.

A-8.2 Determine the organically combined sulphates (SO_3) existing as neutralized sulphonic esters as in **A-8.2.1**.

A-8.2.1 Weigh about 2 g of the sample into a platinum dish and add 25.0 ml of 0.1N sodium hydroxide. Evaporate the mixture to dryness on the water bath and heat in an oven at 100-105°C for 1 h or longer if slow drying. Then ignite by placing the basin on a pipe clay triangle in a muffle furnace at a dull red heat (500-550°C) for 30 min. Remove the basin from the muffle furnace, allow to cool and add 5 ml of 30 percent hydrogen peroxide to oxidize any bisulphite formed and heat in a muffle furnace again (350°C) for 30 min. Remove the basin from the muffle, allow to cool and thoroughly wash out with hot distilled water into a beaker. Add 25 ml of 0.1 N sulphuric acid (H_2SO_4), boil the solution down to about 50 ml to expel carbon dioxide, and allow to cool. Titrate with 0.1 N sodium hydroxide using five drops of 0.1 percent methyl orange as indicator until the colour turns yellow.

A-8.3 Calculation

A-8.3.1 Calculate the organically combined sulphates (SO_3) existing as neutralized sulphonic esters M as follows:

First step:

$$T = \frac{(X - Y)}{W}$$

where

T = resultant acidity expressed as milligrams equivalent sodium hydroxide per 10 g of oil;

X = milligrams equivalent alkali used = $(25 \times N_l)$ + (titre value × N_I), where N_1, is the normality in N of sodium hydroxide solution;

Y = milligrams equivalent acid used $= (25 \times N_2) + VN_2$

where

V = volume in millilitres of any additional sulphuric acid added;

N_2 = normality of sulphuric acid; and

W = weight of the sample, in g.

Second step:

$M = (T - F)$ milligrams equivalent per 10 g of oil

$= (T - F) \times 0.800\ 66$ percent.

NOTES

1 In case of ammonia, neutralized oils

$M = (T - F - T_1)$ milligrams equivalent per 10 g of oil

$= (T - F - T_1) \times 0.800\ 66$

where

T = resultant acidity expressed as milligrams equivalent sodium hydroxide per 10 g of oil (*see* First step);

F = increase in acidity in milligrams equivalent per 10 g of oil (*see* **A-7.4.1**);

T_1 = milligrams equivalent ammonium salts per 10 g of oil and is determined as follows:

2 g of sample is boiled with 50 ml of 0.1 *N* alcoholic KOH and the ammonia distilled into 100 ml of saturated boric acid solution containing the indicator (mix indicator solutions of 0.06 percent methyl red and 0.04 percent methylene blue in 96 percent ethyl alcohol and take 2 ml of the mixed indicator solution in 1 litre of water). The distillate is treated with 0.2 N hydrochloric acid:

$$T_2 = \frac{VN}{W} \times 10$$

where

V = volume of hydrochloric acid, in ml;

N = normality of hydrochloric acid; and

W = weight of the sample, in g

2 If T and F are negative, retain the sign in the equations.

A-8.3.2 Total organically combined sulphates (SO_3) expressed as milligrams equivalent per 10 g of oil consists of the sum of the percentage got in **A-7.4.1** and **A-8.3.1**.

A-9 DETERMINATION OF TOTAL ASH

Weigh 3 g of oil in a platinum dish and heat gently until a charred residue remains. Extract this with hot water to remove soluble salts, filter through a low ash filter paper and wash thoroughly. Ash the filter paper and charred residue, moistening with ash-free hydrogen peroxide, if difficulty is experienced. Allow the dish to cool, add the aqueous extract and evaporate on a steam bath. Finally complete the ashing at a low temperature as possible. Cool the dish and weigh.

A-10 DETERMINATION OF PENTACHLOROPHENOL (PCP)

A-10.1 Transfer about 5 g of the sulphated oil into a separating funnel of about 250 ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent *w/v* aqueous solution and ensure that the *p*H of the contents is acidic. Then carry out liquid — liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note). Transfer the aqueous layer into another separating funnel and carryout three more extractions of the aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps explained in **4.3.4.2** of IS 14575.

A-10.2 Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evaporator.

A-10.3 The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Leather, Tanning Materials and Allied Products Sectional Committee, CHD 17

Organization	*Representative(s)*
Central Leather Research Institute, Chennai	Director (***Chairman***)
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

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